



Size effect in microcompression of polystyrene micropillars



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ABSTRACT

Experiments by He et al. [J.Y. He, Z.L. Zhang, M. Midttun, G. Fonnum, G.I. Modahl, H. Kristiansen, and K. Redford. Size effect on mechanical properties of micron-sized PS-DVB polymer particles. *Polymer* 2008, 49(18), 3993] have indicated that polymers exhibit enhancement of strength at length scales of the order of microns. This is surprising since size effects, widely reported for FCC metals, owe their origin to the presence of crystalline defects, particularly dislocations. On the other hand, polymers being a material of choice in microfabrication, these results assume technological importance. In this work, we conduct controlled experiments on polystyrene (PS) micropillars to ascertain whether they exhibit size effects. The pillars are microfabricated and hence free of beam damage. The experimental load displacement curves obtained from microcompression experiments are matched with computer simulations on similar samples using a well-calibrated constitutive model. Our results demonstrate significant intrinsic size effect on the yield strength, though effects of size on rehardening response turned out to be an experimental artifact.

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1. Introduction

With rapid miniaturisation of engineering components, we come across number of situations where the strength of a material is found to be a function of the external geometric size of the sample. Possibly the most striking example of this has been demonstrated for nanopillars of single crystalline Au in compression [1], where not only was the strength determined to be many times higher the bulk material, the stress strain curve was populated with intermittent strain bursts indicating a significantly different deformation mechanism. Subsequently, amplification of strength at small scales have been reported for a number of FCC single crystalline materials (eg. for Ni in Ref. [2], Cu in Ref. [3] and Al in Ref. [4]; also, see Ref. [5] for a review). In fact, Dou and Derby [6], based on the experimental data available for uniaxial compression tests on micro and nanopillars of Au, Cu, Ni and Al have proposed a scaling law of the form $\tau_{res}/G \sim (d/b)^m$ (where τ_{res} is the resolved shear stress, G the shear modulus, d the diameter of the sample and b the magnitude of the Burger's vector) with $m \sim -0.6$. Size effects, albeit less pronounced than in compression, have been reported on uniaxial tension as well (eg. Refs. [7,8]).

Uchic et al. [5] have critically discussed effects of sample

preparation methodology via the Focussed ion beam (FIB), aspects of the testing procedure, and sample geometry on nano and micropillar compression experiments on FCC single crystal materials. There seems to be a clear consensus that size effects in these materials is not an experimental artifact and is intimately tied to the initial dislocation microstructure prevailing in the sample [9].

More recently, size effects have been observed in samples that are not single crystalline (as well as in non-FCC single crystalline materials though we do not discuss them here; see Ref. [10] for a review). These reports include bicrystalline Al micropillars containing a single grain boundary [4,11], nanograined materials [12] and nano-laminates [13]. Results in these materials are less unequivocal. Compression experiments on nanograined Ni-W nanopillars show a decrease in strength with size [12]. Experiments on pillars with a single grain boundary show contradictory trends among the various reported results.

In amorphous materials, where the dislocation based explanations for the size effect do not apply, experimental observations of size effect have been confined mainly to metallic glasses. In these materials, like in crystalline ones, the samples are made by FIB milling, with the smallest reported diameter being 150 nm [14]. However, there seems to be no consensus on how size effects manifest in these materials. Large increase in yield stress over the bulk material has been reported by Lai et al. [15] and Lee et al. [16] for Zr and Mg based metallic glasses respectively. On the other hand, Volkert et al. [17] reported a decrease in strength of PdSi

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micropillars with diameter. Chen et al. [18] have compiled experimental results for the strength of Cu and Zn based metallic glass micropillars with diameter, which shows that the measured yield stress is almost invariant with size. At the same time, Chen et al. [19] also note that, as the pillar diameter decreases, the apparent deformation mode seems to become more homogeneous. Interestingly, the stress strain curve records more frequent shear banding events in the case of smaller pillars but appears to be more homogeneously deformed under microscopic observation. Several authors have also pointed out that experimental artifacts like specimen taper and ion beam damage obfuscate the interpretations of compression tests in micropillars of metallic glasses.

Polymers form an important class of amorphous materials. But studies on the effect of specimen size on the strength of these materials, unlike amorphous metals, is rarely made. There are indications however, that significant increase in strength of amorphous polymers occur with decrease in size. He et al. [20] have performed diametral compression experiments on micron-sized spheres (~2.5 to ~25 μm in diameter) of polystyrene-divenylbenzene (PS-DVB). Their results show that the stress at 4% strain increases by almost 50% as the diameter of the spheres decrease from 25 to 2.5 μm . Wang et al. [21] have conducted compression tests on FIB milled epoxy micropillars and reported significant increase in yield and fracture strength with decrease in pillar diameter from about 5 μm to sub-micron ranges. The size effect on the yield strength has been attributed to the lack of weak bonds in smaller samples. As in the case of metallic glasses, probability of yielding of the micropillar is modelled with Weibull statistics, with the yield strength in compression $\sigma_0 \propto \varphi^{-\nu}$ (φ is the pillar diameter) with $\nu \approx 0.9$.

In the present work, amorphous, glassy PS micropillars have been fabricated by hot embossing rather than by FIB milling. Cylindrical micropillars with negligible taper, ratio of length to diameter of nearly 2:1 and diameters ranging from 38 to 190 μm are fabricated using the technique described in Section 3.1. The surfaces of these pillars are free of damage from processing issues unlike the ion beam case.

The micropillars are loaded in compression and their force displacement curves are noted. In order to ascertain whether “smaller is stronger” applies to PS, we fit, through Finite Element (FE) simulations, the parameters of a well established constitutive model against experimentally obtained force displacement response of the micropillar. Size effects can be assumed to exist if the parameters in the constitutive model need to be revised in order to fit the force displacement curves of pillars with different diameters. It should be noted that constitutive models for polymers typically contain a larger number of parameters than those for metals, but we are especially interested in the small set that governs the yielding. If these parameters turn out to be sensitive to the diameter, size effects exist. Our results indicate that yielding in PS is indeed affected by the size. Moreover, apart from yielding, there are other aspects of the stress strain behaviour that are also affected by size. We have performed careful FE simulations to separate extrinsic and intrinsic size effects in PS. In the light of the increase need of building microscale devices with polymers the results discussed below have a high level of significance.

2. Constitutive model for PS

When crazing is suppressed, as in uniaxial compression, glassy amorphous polymers generally show a linear visco-elastic behaviour at small strains, followed by yielding and a significant ‘yield drop’ (i.e. a significant difference between an ‘upper’ and ‘lower’ yield points), almost perfectly plastic deformation and finally, steep ‘rehardening’. Several constitutive models for glassy amorphous

polymers exist [22–25] that successfully describe this basic behaviour. The models differ in their ability to describe more subtle aspects of the deformation like the rate hardening behaviour, unloading behaviour, thermo-mechanical response etc. These aspects are not of much significance to our work. Hence, we use the Wu and van der Giessen [23] model, without further justification, to model the micropillar experiments. Only a brief description of the essential features is given here. Details about the model [23] and methods of fitting the parameters [26] are available elsewhere.

The constitutive model makes use of the decomposition of the rate of deformation \mathbf{D} into an elastic (\mathbf{D}_e) and a plastic (\mathbf{D}_p) part as $\mathbf{D} = \mathbf{D}_e + \mathbf{D}_p$. Prior to yielding, no plasticity takes place and $\mathbf{D}_p = 0$. In this regime, most amorphous polymers exhibit visco-elastic effects but these are neglected here since we are primarily interested in the effect of the bulk plasticity. Assuming the elastic strains to remain small, the constitutive model takes the form,

$$\overset{\nabla}{\boldsymbol{\sigma}} = {}_e : \mathbf{D}_e, \quad (1)$$

where $\overset{\nabla}{\boldsymbol{\sigma}}$ is the Jaumann rate of the Cauchy stress given in terms of the spin tensor \mathbf{W} as

$$\overset{\nabla}{\boldsymbol{\sigma}} = \dot{\boldsymbol{\sigma}} - \mathbf{W}\boldsymbol{\sigma} + \boldsymbol{\sigma}\mathbf{W}, \quad (2)$$

and ${}_e$ the usual fourth-order isotropic elastic tensor given by

$${}_e = \lambda \mathbf{I}_2 \otimes \mathbf{I}_2 + \mu \mathbf{I}_4. \quad (3)$$

Here, λ and μ are Lamé’s constants, while \mathbf{I}_2 and \mathbf{I}_4 are the symmetric second and fourth order identity tensors. Assuming that the yield response is isotropic, the isochoric visco-plastic strain rate

$$\mathbf{D}_p = \frac{\dot{\gamma}_p}{\sqrt{2}\tau} \bar{\boldsymbol{\sigma}}', \quad \text{with } \tau = \sqrt{\frac{1}{2} \bar{\boldsymbol{\sigma}}' : \bar{\boldsymbol{\sigma}}'}, \quad (4)$$

is specified in terms of the equivalent shear strain rate $\dot{\gamma}_p = \sqrt{\mathbf{D}_p : \mathbf{D}_p}$, the driving stress $\bar{\boldsymbol{\sigma}} = \boldsymbol{\sigma} - \mathbf{b}$ and the related equivalent shear stress τ . The back stress tensor \mathbf{b} describes the progressive hardening of the material as the strain increases and will be defined later. The equivalent shear strain rate $\dot{\gamma}_p$ is taken from Argon’s expression [27].

$$\dot{\gamma}_p = \dot{\gamma}_0 \exp \left[\frac{-As_0}{T} \left\{ 1 - \left(\frac{\tau}{s_0} \right)^{5/6} \right\} \right] \quad \text{for } T < T_g, \quad (5)$$

where $\dot{\gamma}_0$ and A are material parameters and T the absolute temperature (note that plastic flow is inherently temperature dependent through Eq. (5)).

In Eq. (5) the so called athermal shear strength s_0 is related to elastic molecular properties in Argon’s original formulation but is considered here as a material parameter. Further, in order to account for the effect of strain softening and for the pressure dependence of the plastic strain rate, s_0 in Eq. (5) is replaced by $s + \alpha p$, where α is a pressure sensitivity coefficient and $p = -1/3 \text{tr} \boldsymbol{\sigma}$. The shear strength s is taken to evolve from the initial value s_0 with the plastic strain rate through

$$\dot{s} = h(1 - s/s_{ss})\dot{\gamma}_p, \quad (6)$$

so as to incorporate strain softening in a simple way. Here, h controls the rate of softening, while s_{ss} represents the final, steady state value of s .

Completion of the constitutive model requires the description of the progressive hardening of amorphous polymers upon yielding due to deformation-induced stretch of the molecular chains. This

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